THE ROLE OF ACCELERATION FACTOR IN PROTON IRRADIATION TESTS

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ABSTRACT

Materials need to undergo qualification testing before being applied to any space mission. Testing includes radiation hardness. It is the aging response of materials to corpuscular and/or electromagnetic radiation. The type of radiation and its amplitude depend on the space environment under study. To perform tests within reasonable times, radiation amplitude must exceed that present in space. Hence, accelerated irradiation tests must be performed.

Our main objective is to emphasise potential risks, which may occur upon performing irradiation tests of satellite components with an acceleration factor higher than one.

Degradation studies of thin metallized foil with an oxide layer were carried out. The irradiation tests were performed by use of a linear proton accelerator at DLR-Bremen for three different proton flux magnitudes.

During the irradiation, the sample surfaces were populated with blisters filled with molecular hydrogen gas. It turns out that the size and the number of blisters depends strongly on the proton flux magnitude. More importantly, we have proven that there exists a threshold of the flux magnitude above which the blistering process is being decelerated. It has direct correlation with the metal oxide layer and its aging condition.

The here presented results are important for planning of proton irradiation tests. Too high proton fluxes lead to an acceleration of material degradation that does not reflect the true nature of aging processes as take place in the environment under study. In our case, the presence of the outer aluminium oxide layer has a decisive role in the aluminium degradation process.

INTRODUCTION

Terrestrial electromagnetic and corpuscular radiation tests of satellite components shall confirm their suitability for application in space. Irradiation experiments can be performed with radiation intensities varying from low to high values, which may significantly exceed those present in space. Frequently, the choice is dictated by economic reasons, i.e. the costs of laboratory time usage.

To obtain the scientific results presented in this work, we have used monoenergetic 2.5 keV protons and thin polyimide films covered at both sides with 100 nm aluminum layers as target material.

Here, we discuss a degradation process which turned out to be very sensitive to the amplitude of incident radiation. It is the so-called hydrogen blistering process. It refers to formation of tiny surface blisters filled with hydrogen molecular gas. The gas is formed by recombination processes of the incident protons bombarding aluminium surface and the electrons of the sample. It has been proven that the process takes place in space under specific sets of environmental conditions (i.e. temperature, proton flux, fluence and energy).

We present the sensitivity of the process with respect to the proton flux. The chosen fluxes are comparable to those prevalent in space. This provides an impression of how the process evolves with different acceleration factors.

The blistering process was studied by analysing the surface morphology parameters after receiving different proton fluxes and fluences. Both the average blister radius and the number of blisters as a function of the exposure time to a certain proton flux have been analysed.

In view of the results presented in this work, which were primarily focused on the hydrogen blistering phenomenon and its sensitivity to process parameters, i.e. flux of incident protons, we have proven that even a small change of the incident radiation flux magnitude can have a big impact on the outcome of laboratory tests. Too high proton fluxes may lead to an accelerated material degradation that does not reflect the true nature of aging processes as present in the environment under study.

ACCELERATED IRRADIATION TESTS

Referring to the ECSS standard [1], an acceleration factor (ACCF) is defined as "ratio of the intensity of a degrading factor applied to a material at the laboratory during a space simulation versus the intensity of the same degrading factor in space". ACCF higher than one means that in laboratory an intensity of the radiation is larger than that corresponding in space. Studying the degradation of materials, one must remember that it is "impossible to reproduce the space environment for ground testing of space system elements because of the variety and complexity of the environments and the effects on materials" [2].

Therefore, the aim must be to establish test conditions that simulate the most degrading effects, i.e. one has to identify those space radiation source which degrade the material under study strongest. Then, a witness sample must be exposed to a radiation intensity which is equivalent to that in space. The sample shall be then examined with respect to its response to the radiation. After that a second sample should be exposed to the same radiation but with larger intensity. The response of this specimen to the radiation is afterwards compared to the first sample. If the results are identical, one can increase the ACCF and perform the test again. Only with such procedure one can guarantee that the accelerated irradiation test reflects the true aging behaviour of a test material to the corresponding radiation in space. In our experiments the proton flux magnitude was identified as the degrading factor to calculate the ACCF.

THE COMPLEX IRRADIATION FACILITY

The accelerated irradiation tests were performed by help of the Complex Irradiation Facility (CIF) at DLR, Bremen [3]. It is an Ultra High Vacuum (UHV) facility equipped with two linear accelerators and three light sources. All working simultaneously are meant to simulate space environment. Corpuscular sources are proton and electron accelerators. The kinetic energy of the particles can be set from 2keV to 100keV while the particle currents can be varied from 1nA to 100µA. Electromagnetic sources are as follows: an Argon-VUV-source, a Deuterium lamp and the so-called Solar Simulator equipped with an arc-Xenon lamp. All, if working together, cover a wide wavelength range from 40 nm to 2150 nm. The samples can be tempered from lN_2 level to almost 400°C. The facility is depicted in the Fig. 1. The protons are generated by ionization of hydrogen gas. The plasma is then pushed to the acceleration section by a proper value of extraction voltage. The protons are speeded up while passing through the acceleration section and guided to the irradiation chamber where a specimen is being placed.

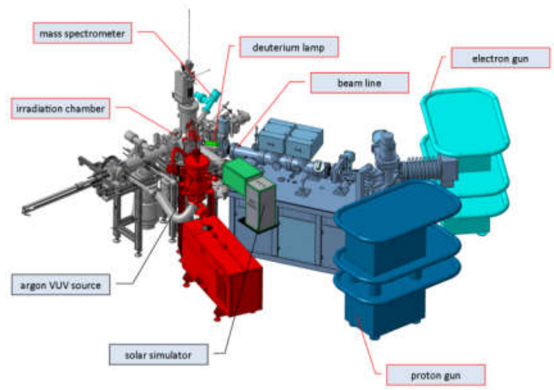

Fig. 1. The Complex Irradiation Facility [4].

PROTON FLUXES IN THE INTERPLANETARY MEDIUM AT 1AU DISTANCE FROM THE SUN

Flux of protons in the interplanetary medium at 1 AU distance from the Sun can be calculated by use of the data recorded by the Sun space observatories, e.g. the Advance Composition Explorer (ACE). The so-called cumulative flux at 1 AU is ~2 x 10^{12} p^+ cm^{-2} s^{-1}. That value was the highest flux recorded in the year 2005. The cumulative flux is defined as follows. ACE records the basic solar wind parameters which can then be used to compute the proton flux spectra. Cumulative flux is a sum of average proton fluxes for a given energy range. In 2005, the range of these energies was from ~0.5 keV to ~5.5 keV.

HYDROGEN BLISTERING – ACCELERATED PROTON TEST

The here presented experimental findings are compressed representation of the test results which are in detail published in [4].

Hydrogen blisters appear on metallic surfaces while being exposed to proton radiation. They are tiny surface pockets filled with hydrogen molecular gas. The gas is formed by recombination processes of the incident protons and the electrons of the sample. It has been proven that this process takes place in space under specific sets of environmental conditions [4, 5].

The test material was a 7.5 µm thick Upilex-S foil covered at both sides with a 100 nm vacuum deposited Aluminium layer. The material was stored under ambient conditions. Therefore, on the surfaces were additional AlOx layers of ~5nm [6].

The samples were mounted onto a sample holder and then inserted into the CIF irradiation chamber. The sample holder was kept during the experiment at constant temperature of 33°C ± 0.22°C and pressure of ≈10^{-8} mbar range. The choice of this temperature level was motivated by our previous experimental studies [5].

The results of the test campaigns are presented in Table 1. First column is the name of a sample, second is the irradiation time, third is the proton fluence, and fourth is the average blister radius of the population.

Table 1 Test campaign results

Sample	Time x 10^5 [s]	Fluence x 10^{17} [p+ cm^{-2}]	<R> [μm]
S1	0.62	1.40	0.112
S2	1.39	2.97	0.136
S3	2.84	6.10	0.154
S4	3.41	7.74	0.126
S5	5.52	11.86	0.132
S6	5.95	13.51	0.090
S7	9.74	20.93	0.095
S8	9.97	22.66	0.099
S9*	9.41	12.81	---
S10*	3.92	12.77	---

An average blister radius of the population was calculated by digital processing of the Atomic Force Microscope (AFM) pictures. The blisters were counted, their radius was measured, and an average value was calculated. This was achieved by adopting the Hoshen-Kopelman algorithm [7].

Comparing the average blister radius of the population and the irradiation time of the specimens, one can recognize that the blister growth evolution can be classified into three stages, see Fig. 2.

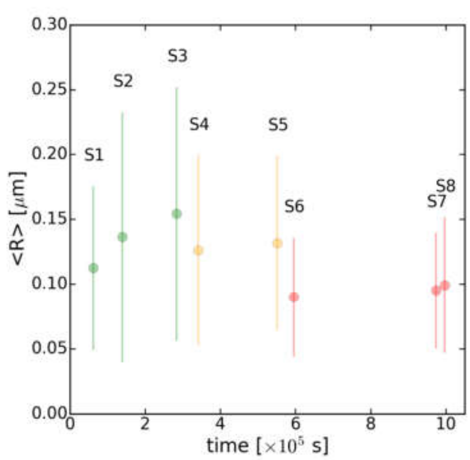

Fig. 2 Average blister radius from the population as function of the irradiation time

Further AFM and Field Emission Microscope (FEM) analysis reveal that the blister growth is determined by the degradation level of the native AlOx-layer [4]. The first stage proceeds without the oxide layer degradation. The recombined hydrogen atoms cluster into blisters and expand the very top layer of the Aluminium. During the second stage, the oxide layer starts to crack allowing the hydrogen to leave the specimen. Hence, blister's size and their number are smaller than at the first stage of the growth. The third stage is when the oxide layer is mostly degraded and the recombined hydrogen can easily diffuse out of the specimen. The number and the size of the blisters are dropping by about ~50% as compared to the first stage of the process. The corresponding AFM pictures of the surface morphology of the samples are shown in Fig. 3. There one can see clearly that the size and number of blisters decreases while the irradiation time increases.

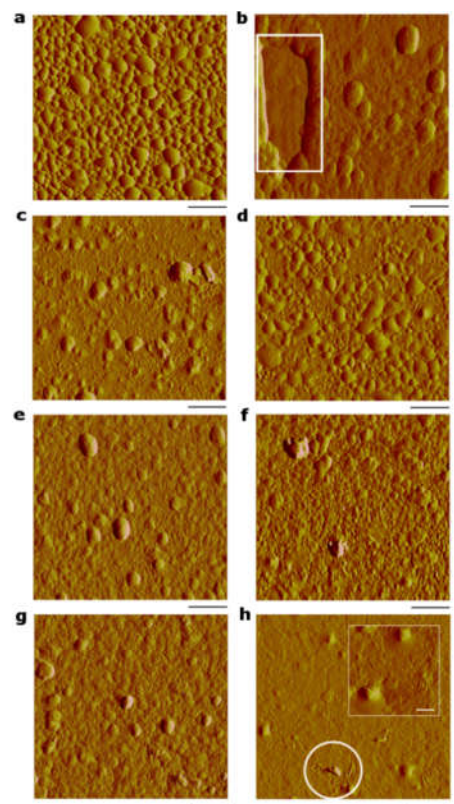

Fig. 3 Surface morphology of the specimens from S1 (a) to S8 (h). Black scale bar equals to 1 μm, white scale bar equals to 0.3 μm.

The samples S1 – S8 have been exposed to the same proton flux of 2.27 x 10^{12} p^+ cm^{-2} s^{-1}. Two additional specimens have been irradiated to the protons with larger and lower proton flux, i.e. Sample S9* to 1.36 x 10^{12} p^+ cm^{-2} s^{-1}, while sample S10* to 3.26 x 10^{12} p^+ cm^{-2} s^{-1}. The fluence of all of the three samples S6, S9*, and S10* is comparable, see Table 1.

Sample S9* has been exposed to ~1.5 times smaller proton flux than present at 1 AU, see Fig. 4. Here, the sample surface was populated with blisters; however the AlOx layer did not crack throughout the irradiated area.

The small dark areas are places where the AlOx layer has been delaminated from the aluminium substrate.

For the sample S6 small delamination centres but also large surface areas delaminated from the aluminium substrate are seen in Fig. 5. One can recognize also cracks of the native AlOx layer.

Sample S10* received the highest proton flux, see Fig. 6. Clearly, the AlOx layer cracks over the whole irradiated area. They are thin dark intersecting lines, covering the sample area exposed to protons. Also a large crack has been spotted at the base of a blister. That area has been enlarged in small rectangular window.

Please note that such structures as broken blisters and delaminated AlOx layer can also be observed on the sample S8 surface. They have been marked with rectangular window and circular mark in subfigure **h** of Fig. 3.

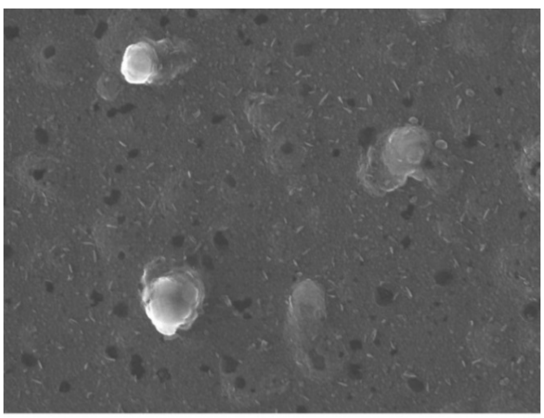

Fig. 4 Small delamination centres are present on the sample S9* surface. No AlOx crack can be spotted. Black scale bar equals to 1 µm.

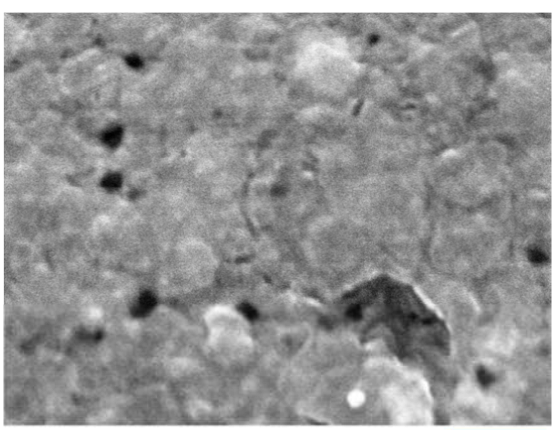

Fig. 5 Delamination centres and cracks of the AlOx layer cover the sample S6 surface. Orange scale bar equals to 0.2 µm.

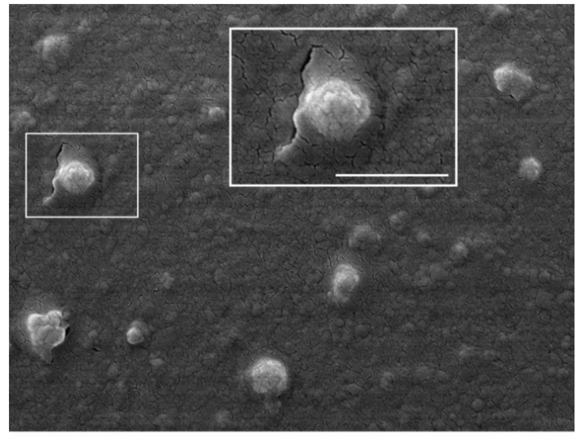

Fig. 6 Cracks of the AlOx layer cover the whole area of the sample S10*. Black scale bar equals to 1 µm, white scale bar equals to 0.5 µm.

CONCLUSIONS

The proton flux magnitude dictates the blister evolution when a native AlOx layer is formed on Aluminium. Such an oxide layer is very likely present on all metallic surfaces exposed to space conditions. It cracks when the flux is 2.27×10^{12} p^+ cm^{-2} s^{-1} or higher. For lower fluxes the native AlOx layer delaminates from its Aluminium substrate as well, but on relatively small areas.

Proton flux received by the here shown samples (S1-S8) represent their state at ~1 AU from the Sun. After only a few days under space conditions the aluminium foil would be covered by hydrogen blisters. A small increase of the proton flux by a factor of ~1.6 causes a significant change in the aluminium surface morphology (see sample S10*). Therefore, the radiation driven blistering phenomenon is very sensitive to process parameters.

REFERENCES

1. ECSS-Q-ST-70-06C, “Particle and UV Radiation Testing for Space Materials”, 2008.

2. ISO15856:2010, “Space systems – space environment – simulation guidelines for radiation exposure of non-metallic materials”, 2010.

3. T. Renger, M. Sznajder, A. Witzke, U. Geppert, “The Complex Irradiation Facility at DLR-Bremen”, *Journal of Materials Science and Engineering A*, vol. 4, pp. 1-9, 2014.

4. M. Sznajder, U. Geppert, M. R. Dudek, „Hydrogen blistering under extreme radiation conditions“, *npj Materials Degradation*, vol. 2, 2018.

5. M. Sznajder, U. Geppert, M. Dudek, „Degradation of metallic surfaces under space conditions, with particular emphasis on Hydrogen recombination processes", *Advances in Space Research*, vol. 56, pp. 71-84, 2015.

6. J. Evertsson et al. "The thickness of native oxides on aluminium alloys and single crystals", *Applied Surface Science*, vol. 349, pp. 826-832, 2015

7. J. Hoshen, R. Kopelman, „Percolation and cluster distribution. I. Cluster multiple labeling technique and critical concentration algorithm", *Physical Review B*, vol. 14, pp. 3438-3445, 1976.